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Continuous porosity characterization: Metric-scale intervals in heterogeneous sedimentary rocks using medical CT-scanner

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1   **CONTINUOUS POROSITY CHARACTERIZATION:**

2   **METRIC-SCALE INTERVALS IN HETEROGENEOUS SEDIMENTARY**

3   **ROCKS USING MEDICAL CT-SCANNER**

4

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13

14 **Abstract**

15 Although computed tomography (CT-Scanning) has been regularly applied to core

16 analyses in petroleum geology, there is still a need to improve our ways to document

17 porosity and porosity distribution in the entire pore scale spectrum, from the tens of

18 nanometer to the meter-scale. Porosity imaging is particularly crucial for complex and

19 heterogeneous rocks such as hydrothermally altered and fractured carbonates. The present

20 work proposes a improved method using medical-CT to reliably estimate reservoir

21 porosity. An in-house core-flooding setup allowed to analyse several individual core

22 samples, scanned simultaneously (dry and saturated), as well as continuous core sections

23 up to 1.5 m long. Without any prior knowledge of samples, three-dimensional alignment

24 and subtraction of the two data sets (dry and saturated states) results in the generation of

25 3D porosity matrices. The methodology tested on a large set of reference core material

26 shows a strong correlation between conventional gas porosimetry techniques and porosity

27 from CT-scan. The added value of the porosity measurements by CT-scan is, first of all,

28 the generation of 3D images of pore network, allowing to assess spatial attributes of

29 macropores, their distribution and connectivity. Secondly, the CT-scan method also

30 provides continuous porosity profile at the millimetric scale. Both developments are

31 crucial for the understanding of reservoir rock properties.

32 **Keywords**: 3D porosity, carbonate, core-flooding, CT scan

33

34 **INTRODUCTION**

35 The spatial distribution of petrophysical properties of reservoir rocks commonly needs to

36 be assessed whether it is for the exploration of hydrocarbons, the identification of

37 efficient reservoir for CO_2 storage or the assessment of geothermal resources (e.g.

38 Kukkonen and Peltoniemi, 1998; Mees et al., 2003; Chadwick et al., 2004; Hartmann et

39 al., 2008; Shi et al., 2009; Goldberg et al., 2010; Perrin and Benson, 2010a). Computed

40 tomography (CT-scanning) has been applied for decades by the hydrocarbon community

41 to supplement conventional core analyses (such as mineralogy, porosity, permeability) or

42 wireline logging interpretation (Vinegar and Wellington, 1987; Wellington and Vinegar,

43 1987; Duliu, 1999; Akin and Kovscek, 2003; Taud et al., 2005; Geiger et al., 2009;

44 Baniak et al., 2013). CT-scanning techniques are non-destructive and offer the possibility

45 to quantify internal structures based on the measurement of X-Rays attenuation

46 coefficients, which depend on the chemical composition and physical density of the

47 materials analysed (Duliu, 1999; Akin and Kovscek, 2003; Cnudde and Boone, 2013).

48 Therefore, CT-scanning provides qualitative analysis when applied to subsurface

49 materials (e.g. heterogeneity, damages, presence of fluids); it can also be used to gain

50 quantitative information on cores such as bulk density, porosity, and fluid saturations

51 (Coles et al., 1991; Cnudde and Boone, 2013). Efforts have also been made to use CT-

52 scanning to understand fluid displacement and relative permeability of core material from

53 reservoirs (Hove et al., 1987; Vinegar and Wellington, 1987; Withjack, 1988), notably

54 because of their importance in oil-recovery processes during production stages

55 (Andrianov et al., 2012; Simjoo et al., 2013; Simjoo and Zitha, 2018). Limitations of

56 medical CT (or conventional CT, as proposed by Ketcham and Carlson (2001)) are well

57 known. The image spatial resolution obtained is relatively low (about 0.5 mm on

58 average), allowing characterization and quantification of the spatial distribution of larger

59 structures only such as burrows, roots, primary or secondary framework pores or

60 fractures. The use of medical CT systems is consequently being complemented by

61 MicroCT and synchrotron based systems (Ketcham and Carlson, 2001; Cnudde and

62 Boone, 2013; Wildenschild and Sheppard, 2013) that offers a better resolution.

63 However, there is a critical need to get data on the entire pore scale spectrum, from the

64 tens of nanometer to the meter-scale in order to build comprehensive datasets for rocks,

65 sediments and soils (Okabe and Blunt, 2007; Biswal et al., 2009; Vaz et al., 2014;

66 Bultreys et al., 2016b; Xiong et al., 2016). Porosity imaging and pore-network modelling

67 techniques are crucial for characterizing complex and heterogeneous rocks such as

68 carbonates (Sok et al., 2010; Pak et al., 2016). Deposition processes and diagenesis result

69 into heterogeneity at multiple spatial scales so that predicting petrophysical properties is

70 particularly challenging in carbonate reservoir rocks. Pore structure affects connectivity,

71 conductivity and permeability that all influence oil recovery mechanisms, a key aspect for

72 economic geology. MicroCT and synchrotron-based systems give access to spatial

73 resolutions of few micrometers to deal with pore-particles interfaces but sample size is

74 restricted to few millimeters (Vaz et al., 2014) and is time-consuming. The trade-off

75 between sample size and spatial resolution (i.e. voxel size), mainly imposed by the micro-

76 CT scanner detector characteristics, is therefore a limitation to upscale data obtained at

77 the pore scale and justifies modelling. Even if significant developments are made

78 regarding small scale heterogeneity characterization techniques, there is a need to

79 optimize characterisation of heterogeneous material such as fractured or dolomitized

80 carbonates at the centimeter scale.

81 Coupled with core-flooding experiments, CT scanning are well establish in reservoir

82 studies to monitor the progress of multiphase displacement inside porous media

83 (Schembre and Kovscek, 2003; Shi et al., 2009). Different core-flooding setups exist,

84 depending on the fluids involved or the experimental conditions, such as the confining

85 pressure that can be radial and/or axial, the temperature (reservoir condition), or the fluid

86 injection mode (Wang et al., 1984; Alemu et al., 2013). The reliability of such scanning

87 technique was verified through a study of oil and gas shows in eastern Québec, where an

88 industry, university and government partnership was developed to improve current CT

89 scan methods, which remained difficult to use when characterizing reservoir properties in

90 heterogeneous carbonates. The authors found, to the extent of their knowledge, that

91 rigorous and complete scientific literature on CT scan technique was needed to

92 implement a detailed characterization program with specific interest to carbonate rocks.

93 Studied rock units were locally found within highly fractured intervals associated with

94 replacive and pore-filing hydrothermal dolomites. The industry partner, Squatex Inc.,

95 drilled and cored over 6000 m of stratigraphic exploration wells and needed an efficient

96 and rapid way to assess the spatial distribution of reservoir properties in the area in order

97 to plan their future works.

98 This project objective was to complement and enhance current CT scan methods applied

99 to sedimentary rocks in order to reliably document porosity distribution and connectivity

100 in heterogeneous carbonate reservoirs and to predict its distribution over a metric scale.

101 An improved methodology combining a core-flooding setup and medical-CT analyses to

102 obtain the porosity of heterogeneous rock material was consequently developed. No

103 confinement pressure is applied during saturation: water flows around sample and the

104 experiment is conducted at room temperature. The methodology has been tested on

105 reference core material that has a wide range of porosity values and includes eight

106 different sedimentary rock types. To test this enhanced CT methodology, porosity results

107 are compared with conventional ways to determine porosity in rock material (i.e. helium

108 gas porosimetry). In addition, the methodology was tested on a specific metric section of

109 an heterogeneous carbonate interval. Within this interval, 3D porosity matrices were

110 generated to visualize the connected porosity network, as well as a continuous porosity

111 profiles at the millimetric scale.

112 **PREVIOUS WORKS**

113   1) CT-scanning

114 Both medical and microfocus CT-scanning (Micro-CT) has been commonly used for

115 decades for core analyses in the Oil and Gas sector in order to analyse porosity, fractures

116 patterns, or assess fluid flow in porous rocks (e.g. Honarpour et al., 1985; Grader et al.,

117 2000; Van Geet and Swennen, 2001; Akin and Kovscek, 2003; Karacan et al., 2003; Van

118 Geet et al., 2003; Denney, 2004; Taud et al., 2005; Geiger et al., 2009). Images generated

119 contains relative density measurements in Houndsfield Unit (HU) and allow qualitative,

120 such as internal structures description, and quantitative information analysis. Different

121 parameters can be derived from HU values as it relates to three physical aspects: real

122 density, chemical composition (atomic number) and porosity. To isolate each properties,

123 different methods has been developed. The atomic number can be computed using Dual-

124 energy scans (Wellington and Vinegar, 1987; Alves et al., 2014; Jussiani and Appoloni,

6

125 2015). Density estimation in g/cm^3 is often estimated using calibration points obtained

126 from core plugs. Finally, porosity is estimated using various approach: (1) segmentation

127 of pores, (2) mixels (or mixed pixels(Kato et al., 2013) assuming grain density and (3)

128 saturation technique and subtraction of saturated and dry state (Withjack, 1988; Davis et

129 al., 1992).

130   2) Core-flooding system

131 From the 1950's, hydrocarbon exploration companies have commonly used X-rays to

132 study reservoir properties (Morgan et al., 1950) and, with the advance of 3D computed

133 tomography, to visualize fluid flow through reservoir rocks, calculate porosity and oil

134 saturation (Vinegar and Wellington, 1987; Wellington and Vinegar, 1987; Withjack,

135 1988). Numerous papers provided excellent syntheses on computed tomography

136 principles and common practices (e.g. Newton and Potts, 1981; Ketcham and Carlson,

137 2001). With respect to core flooding experiments, tests under CT are primarily made to

138 observe fluids displacement and to gain information about relative permeabilities

139 (e.g.Wang et al., 1984; Hove et al., 1987; Wellington and Vinegar, 1987; Soltani et al.,

140 2009), either comparing two liquid phases (e.g. oil, brines) or using a gas phase (e.g.

141 CO$_2$) and a denser liquid phase (Schembre and Kovscek, 2003; Shi et al., 2009; Perrin

142 and Benson, 2010b; Alemu et al., 2013; Krause et al., 2013; Krause and Benson, 2015;

143 Jackson et al., 2018). In more recent years, core-flooding studies were carried out with

144 one sample at the time only and were usually associated with a dynamic set-up, using

145 pump(s) to ensure constant fluid circulation trough the core holder and samples. Most

146 common setup involves a water filled chamber made of multi layers sleeve and hosting

147 the sample. The chamber is maintained at particular confining pressure and temperature

148   in order to reproduce reservoir conditions at depth (Perrin and Benson, 2010a; Krevor et

149   al., 2012; Pini et al., 2012).

150   In the late 1980's, Withjack (1988) presented a protocol to measure saturation and

151   porosity with a core-flooding device under CT. This work was based on X-rays and

152   porosity determination principles recognized years before (Morgan et al., 1950; Laird and

153   Putnam, 1959). The setup of Withjack (1988) first involved core samples placed in an

154   aluminium chamber and scanned in a dry state. The aluminum chamber was used to

155   remove lower-energy photons and limit beam hardening artefacts. Prior to the

156   experiment, polyethylene bottles filled with sodium iodide solutions were scanned

157   separately inside the core holder. Then core samples were removed from the chamber, put

158   under vacuum conditions and immerged in the sodium iodide solution. Once samples

159   were considered saturated, they were put back in the chamber and scanned under CT.

160   Withjack (1988) tested a Berea sandstone and a dolomite with both porosity around 20%.

161   Porosity was also measured by the re-saturation method for comparison purposes.

162   Although CT-scan capabilities remained limited at that time, Withjack (1988)

163   demonstrated that CT-scan porosity values correlated well with those obtained by re-

164   saturation ($\pm$ 1 %). The approach described in this paper is primarily based on the so-

165   called X-ray saturation technique developed by Withjack (1988) , but includes recent

166   advances such as procedures for registration and to reduce image noise (e.g. Ketcham and

167   Iturrino, 2005; Pini et al., 2012; Pini and Madonna, 2016).

168   3) Doping agents used with CT-scan

169   A doping agent is a fluid with dissolved salts having a high atomic number ($Z \geq 50$),

170   which alters X-rays absorption properties of the fluid phase. This enhances the contrast

171 between solid and fluid phase (Wildenschild et al., 2002). The use of such dopant is

172 common in the hydrocarbon industry when working on multiphase fluid flow

173 experiments (e.g. Vinegar and Wellington, 1987), and the most popular choices for

174 doping brines are sodium bromide (NaBr), sodium molybdate (Na_2MoO_4) and sodium

175 iodide (NaI). Doping agents are also widely used in other research areas such as soils

176 science (Hopmans et al., 1992; Anderson et al., 2003; Helliwell et al., 2013; Vaz et al.,

177 2014).

178 In the frame of this paper, we present an improved methodology in a sense that we

179 combine, as simple as it can, a core flooding system that does not require any

180 confinement. In addition, the setup allows the saturation of multiple meters of samples

181 simultaneously, thus minimizing the operating cost. In order to validate the methodology,

182 we studied an extended range of porosity (~1% up to ~30%) and different sedimentary

183 rock types, including sandstone but with specific interest to dolomite and limestone.

184 **METHODOLOGY**

185   1) Samples

186 1-1 Reference samples

187 Reference core materials were obtained from two distinct vendors located in the USA:

188 Cleveland Quarries (Vermilion, Ohio) and Kocurek Industries Inc (Caldwell, Texas).

189 Eight different lithologies commonly used as test material in the hydrocarbon industry or

190 rock-mechanics studies were selected (e.g. Churcher et al., 1991; Devarapalli et al., 2017;

191 Islam et al., 2018). These lithologies were chosen to cover a large range of porosity (1.5

192 to 28 %; Fig. 1; Tab. 1). All reference core samples are cylindrical and diameter is either

193   3.8 or 4.5 cm. For this work, tested variables therefore include lithology type, porosity

194   range, core diameter and length.

195   The Berea sandstone is medium-grained, Mississippian in age and outcrops in Ohio

196   (USA). It is mainly composed of quartz and feldspar with small clay content. Particles are

197   well sorted and subangular with quartz overgrowths. Berea sandstone is probably the

198   most commonly used rock as an analogue for hydrocarbon reservoirs (Pini and Madonna,

199   2016). It has a porosity of approximately 20% (Winkler, 1983; Churcher et al., 1991;

200   Hart and Wang, 1995; Boon et al., 2017). The Nugget Sandstone is fine-grained,

201   laminated, Jurassic in age. This sandstone outcrops in Utah and Wyoming (USA). Its

202   porosity can reach up to 25% (Lindquist, 1988) and it is essentially composed of angular

203   to subangular quartz grains (Fig. 2A). The Boise sandstone is a medium to coarse-grained

204   Late Miocene sandstone (Winkler, 1983) from Idaho (USA). This sandstone is poorly

205   sorted and composed of quartz and feldspar, with minor clay (Fig. 2B). It has a porosity

206   of approximately 28-30%. The Scioto sandstone is homogenous, fine-grained, Missippian

207   in age and outcrops in Ohio (USA). Quartz grains are subangular (Figs. 2C-D). This

208   sandstone commonly has a porosity of approximately 12% (Holder et al., 2001; Bose et

209   al., 2014). The Indiana limestone, also known as the Salem Formation or the Bedford

210   limestone, is a middle Mississippian marine bioclastic carbonate that crops out in south-

211   central Indiana (USA). The limestone is heterogeneous, grainstone to packstone and is

212   mostly composed of calcium carbonate with only little amount of magnesium carbonate.

213   Fossil fragments include bryozoans, echinoderms, brachiopods (Churcher et al., 1991).

214   The Indiana limestone is relatively well cemented and has a porosity of approximately

215   13% (Musselman, 1967; Schmidt and Huddle, 1977; Hart and Wang, 1995; Boon et al.,

216 2017). The Carthage marble, the commercial name for Carthage limestone or Burlington

217 limestone, is a homogenous well cemented crinoid-rich Mississippian limestone (Fig. 2E)

218 that outcrops in Missouri (USA). It is composed predominantly of calcium carbonate with

219 little amount of glauconite. This limestone exhibits low porosity value, usually about

220 1.5% (Musselman, 1967; Martin, 1968). The Silurian Dolomite, is a homogeneous fine

221 grained dolostone in Ohio (USA). It is composed predominantly of non planar dolomite

222 crystals arranged in mosaic (Fig. 2F) and its porosity is usually around 20%, but could be

223 down to 14% (e.g. Islam et al., 2018). The Guelph dolomites, also known as the Baker

224 Dolomite is a homogeneous dolomitized carbonate sand, formed in marine shallow water

225 environment. Even though two types of Guelph dolomites exist, the samples used in this

226 work are fine-grained, light gray dolomite (Churcher et al., 1991). The Silurian Guelph

227 dolomites is present in Ohio (USA) and Ontario (Canada). Its porosity value is usually

228 around 7% but can be much higher (up to 24%).

229 1-2 Silurian core samples

230 Silurian core samples being studied for this paper come from the Gaspé Belt in Québec

231 (sensu Bourque et al., 1995) (Fig. 3). It primarily consists of Upper Ordovician to Middle

232 Devonian fine to coarse nearshore to deep marine clastic deposits with subordinate

233 shallow- to deep-water carbonate platform deposits. The industry partner, Squatex Inc.

234 has drilled several stratigraphic wells over the past ten years. Promising reservoir

235 intervals are located in the cyclic offshore – peritidal carbonates of the lower Silurian

236 Sayabec Formation and the underlying shoreface clastics of the lower Silurian Val-

237 Brillant Formation (Lavoie and Bourque, 2001; Lavoie and Morin, 2004). Even if these

238 two units are not hydrocarbon producers, both have ubiquitous evidence of at least

239   locally, hydrocarbon charge (Lavoie et al., 2009; Dietrich et al., 2011). Hydrothermal

240   dolomites of the Sayabec Formation are found in the well bedded intertidal to shallow

241   subtidal facies (Lavoie and Morin, 2004); they host an exhumed oil field a few kilometers

242   to the east of the study area (Fig. 3) with bitumen and dead oil filling matrix porosity and

243   fractures (Lavoie and Chi, 2010). While drilling in the Témiscouata area (Fig. 3), oil or

244   gas shows were associated with naturally fractured hydrothermal dolomites (HTD)

245   intervals within the Sayabec Formation as well as with the underlying sandstone in the

246   Val-Brillant Formation. In both cases, the shows seemed to be associated with fractured

247   intervals (pers. comm., S. Larmagnat, 2015).

248   Silurian samples were recovered from 1 7/8 inch (47.625 mm) diameter drilled cores. All

249   samples were taken from a specific hydrothermal dolomite interval (Fig. 4A),

250   corresponding to a section of 1.8 meters long. Due to the lack of integrity of some

251   portions, only 18 cylindrical core samples with a minimum length of 5 cm have been

252   selected. Out of this 18 samples set, five samples were sent for helium porosimetry

253   measurements at an external lab, AGAT Laboratories, after the core-flooding experiment

254   was completed (Fig. 4B). Those samples were selected on the basis of representability of

255   the whole 1.8 meter succession. Hence we have selected visually non-porous limestone

256   muds, limestone mud with some visible pore space, vuggy dolostone, fractured dolostone

257   and vuggy and fractured dolostone to cover the entire spectrum of depositional/diagenetic

258   elements present in those carbonates.

259   2) Helium porosity measurements

260   3-1 Reference rock samples

261   Because reference rock samples were not provided with exact porosity values from the

262   core vendor itself, all reference samples were sent to the external laboratory, namely

263   AGAT laboratories in Calgary (Canada), to get helium gas porosity measurements.

264   AGAT Laboratories are private, independent, and routinely run petrophysical properties

265   analyses (including helium gas porosimetry) for the oil and gas industry, academy and

266   governmental research teams (e.g. Connell-Madore and Katsube, 2007; Black, 2014;

267   Gasaway et al., 2018). In addition, depending on sample diameter and length, reference

268   rock samples were analysed by two additional gas porosimetry instruments at INRS.

269   From a total of 30 samples, ten samples have been analysed for gas porosimetry by three

270   distinct instruments and the CT-scanner. All reference samples were analysed at least by

271   one gas porosimeter and the CT-scanner. In all cases, gas porosity measurements were

272   conducted on samples before analysing them under the CT-scan.

273   Gas porosity measurements were obtained from instruments relying on Boyle's law: the

274   pressure exerted by a mass of helium gas is inversely proportional to the volume of the

275   samples. Measuring the change in helium pressure gives the grain volume. Therefore,

276   porosity value results from two measurements, grain volume and bulk volume. The first

277   is measured by the instrument itself and the second is obtained manually using a caliper.

278   Assuming a perfect cylindrical geometry, a caliper is used to measure the length and

279   diameter, and calculate the sample bulk volume. For this study, the calculation of the

280   porosities using the three different instruments have been made using a single bulk

281 volume for each sample in order to minimize potential discrepancies that could arise from

282 three different manual caliper measurements.

283 A total of 30 samples were thus sent to AGAT Laboratories. Prior to analyses, samples

284 are dried in a convection oven for 48 h at 108°C. The helium pressure is set at 2.76 MPa.

285 Porosity values were given a margin of error of +/- 0.005 (0.5 %). Hereafter this

286 equipment will be referred as in-house AGAT gas porosimeter (IHAP).

287 A total of 20 porosity measurements were made using the Core Test Systems AP-608 Gas

288 permeameter-porosimeter available at INRS "Laboratoire ouvert de géothermie" (LOG;

289 Québec, Canada). Prior to analyses, samples were first dried at 108°C for at least 48 h,

290 using a Thermolyne oven (Thermo Scientific). Initial helium pressure is set at 1.38 MPa.

291 In order to examine the reliability of analysis results, each core plug was analysed three

292 times and the results were averaged. This procedure delivered porosity results with an

293 average standard deviation of 0.18. Only 20 reference samples were analysed because of

294 diameter and length restrictions associated with the AP-608 instrument (Tab. 1-2).

295 Few samples were also analysed at the INRS laboratory for Decontamination and Waste

296 Reclamation, using a gas pycnometer (Micromeritics AccuPyc 1330). Routinely used to

297 obtain the density, the device uses the Boyle's Law to measure grain volumes. The

298 instrument is fully-automatic and makes three consecutive runs for each analysis. Only 10

299 reference samples were analysed because of diameter and length restrictions associated

300 with this instrument (Tab. 1-2).

14

301   3-2 Silurian core samples

302   Silurian core samples with their 1 7/8 inch (47.625 mm) diameter could only be analysed

303   by the IHAP at AGAT Laboratories. Out of the 18 cores from the hydrothermal dolomite

304   interval (Fig. 4), five samples were sent to AGAT Laboratories, after the core-flooding

305   experiment was completed. These five samples were chosen to represent the natural

306   heterogeneity of the interval. A detailed description of all five sample is given (Figure

307   4B).

308   3)   Core-flooding experiment

309   4-1 System

310   An in-house core-flooding system was designed to accommodate different sample sizes

311   (diameter and length) and to control experimental variables. The system was built to be

312   simple, reliable and cost effective (Fig. 5). One (or several) horizontal chamber, made of

313   polyvinyl chloride (PVC) or acrylic (plexiglass), an X-ray-transparent material, is

314   connected to two pumps and a water tank. The core holder inside diameter is 41.7mm and

315   50.8mm for $1^{1/2"}$ and $1^{7/8"}$ samples, respectively. Between chambers, Swagelok quick

316   connects (valves) are used because they allow minimal air inclusion and minimal

317   spillage. The laboratory vacuum pump (Welch, WOB-L Pump 2585) is used to adjust and

318   monitor the vacuum level. A diaphragm pump (SHURflo, 2088 serie) is used to ensure

319   constant fluid circulation through the chamber at a flow rate 19.28 l/min. Within the

320   chamber, the internal fittings use customized 3D printed PLA (polylactic acid) core

321   holders to increase stability and create space for water flow around the samples,

322   particularly when the chamber is flooded. For the purpose of this work, core-flooding was

323   performed either with distilled water or sodium iodide solution. All reference samples

324 (n= 30) were saturated using distilled water. In addition, 15 samples (see Tab.4) were

325 saturated using sodium iodide salt (NaI). NaI was chosen because its behaviour is similar

326 to NaCl with respect to argillaceous minerals (Withjack, 1988). In addition, it has a

327 reasonable cost, approximately 170$ per 100g, and can be handle safely without causing

328 hazards.

329 4-1 Protocol

330 Core-flooding experiments were conducted at room temperature and no confinement

331 pressure was applied (workflow is summarized in Fig. 6). Prior to scanning, samples

332 were dried at 108°C for at least 48 h, using a Thermolyne oven (Thermo Scientific). In

333 agreement with Ketcham and Iturrino (2005) , a two-stage scanning protocol was used.

334 Samples were placed in the sealed chamber and first scanned in a dry state. Secondly,

335 vacuum is applied for 24hours, degassing distilled water and samples simultaneously.

336 Then the chamber is flooded with either water or NaI solution (15 g/L), during which the

337 diaphragm pump guarantees constant fluid circulation. This removes air bubbles that

338 could be created during the saturation process, thus maximising fluid contact and

339 reducing the number of connected pores that would not be saturated otherwise. It also

340 constantly provides degassed water at sample-water interface thus extracting residual air

341 bubbles. The use of NaI doping agent, with a concentration of 15 g/L, increases contrast

342 by 30% when compared to water. Such improvement can be crucial when investigating

343 small pore size that falls below the range of medical CT resolution. Core samples were

344 weighted before and after saturation with either water or NaI to evaluate the performance

345 of the saturation process. Sample weight was measured with a Sartorius top-loading

346 balance (CP4202S) having a 0.01 g accuracy. According to the Liquid saturation

347 technique (API, 1998), pore volume can be calculated using bulk volume and known

348 fluid density (water and NaI).

349   4) CT-scan measurements

350 CT measurements were performed using a Siemens SOMATOM Definition AS+ 128 at

351 INRS-ETE. The X-ray tube of the CT scanner was operated at a voltage of 140 kV and a

352 current of either 700 or 350 mAs for reference samples and core samples of the study

353 area, respectively. The same voltage for all CT acquisition was used in order to obtain

354 comparable HU values. However, the current had to be adapted to minimize downtime

355 due to X-ray source cooling. Longer core holder, such as the one used for the Silurian

356 carbonate interval, allowed more samples to be processed simultaneously. The source

357 current was then reduced from 700 mA to 350mA, thus using less power and generating

358 less heat. Downtime between scans, using high X-ray source power, can be more than 15

359 minutes, adding hours of waiting time thus increasing significantly the operating cost. An

360 H70h convolution kernel was used for the reconstruction of the images. The thickness of

361 each CT slice was set to 0.6 mm (Tab. 3). Images were recorded in DICOM format and

362 visualizes with the open-source software Fiji (Schindelin et al., 2012).

363   5) Noise reduction

364 Density changes associated with the infiltration of water can be subtle when porosity is

365 low. In such cases, image noise is problematic and could outweigh the density variation

366 associated with the water saturation. The Pini and Madonna (2016) approach was

367 therefore adopted here to examine how the level of noise changed when averaging several

368 scans or decreasing the resolution, and how this ultimately affected the porosity

369 calculation (Fig. 7). The scan repetition for this study was set to three in order to get a

370 short acquisition time with a low image noise from this method.

371   6) Beam hardening

372 The beam hardening is a common artefact caused by the absorption of low energy photon

373 at sample frontier thus "hardening" the beam by making the mean energy higher. This

374 phenomena, linked with the polychromatic nature of the X-ray spectra emitted, produce

375 density under-estimation at sample center (Ketcham and Carlson, 2001). A software

376 beam-hardening correction to detector readings is applied by the scanner and is optimized

377 for human body, which mainly consist of water. This correction does not remove the

378 artefact due to rock samples. Ketcham and Itturino (2005) have showed that the sample

379 geometry changes the beam hardening profile thus a calibration wedge with a similar

380 density and diameter is required. The strategy to minimize the beam hardening profile

381 variation from dry to saturated was to build a core holder with an internal diameter close

382 to the sample diameter. This creates a nearly identical geometry between dry and

383 saturated state, thus producing a near equivalent beam hardening profile. Moreover, the

384 image subtraction applied at the next stage minimizes the influence of beam hardening.

385   7) Data analysis

386 Data analysis is based on the X-ray saturation technique (Withjack, 1988) but also

387 includes recent developments (Ketcham and Iturrino, 2005; Pini et al., 2012; Pini and

388 Madonna, 2016). Algorithms applied aim at determining porosity by comparing CT

389 images in a saturated state and unsaturated state. The working hypothesis considers a

390 voxel as a mixel that is a mixture of porosity and solid phase material. Density value of

391 one mixel is therefore an average value of its content. When saturating the samples with

392   water or NaI, the connected pores, initially filled with air, is filled with the liquid phase.

393   Since solid phase density and quantity do not change, recorded changes in voxels density

394   are interpreted as the results of pore filling. Equations below (1) summarize the

395   calculation of porosity from density matrices acquired (D).

396   $$\%_{porosity} = \frac{D_{dry} - D_{saturated}}{D_{gas} - D_{fluid}} \qquad (1)$$

397   The calculation is applied for each voxel containing the sample. Grain density is not

398   needed to evaluate porosity since only fluid density in pores changes (Boespflug et al.,

399   1994). Fluid density is known as part of CT calibration ($D_{gas} = D_{air} = -1000$ HU; $D_{fluid} =$

400   $D_{water} = 0$ HU), while the density of the NaI solution in Hounsfield unit was obtained by

401   in situ calibration ($D_{NaI} = 324$HU, for a 15g/L NaI solution).

402   The subtraction of the saturated vs dry data to calculate the porosity was performed using

403   MATLAB®. Prior to subtraction, data registration was performed for each analysis using

404   intensity-based image registration algorithm (MathWorks, 2018). The 3D matrix resulting

405   of this subtraction allowed to visualize the effective pore network and the evaluation of

406   the porosity distribution using statistics. In some instance, a circular binary mask has

407   been used to include large vugs located at sample surface. The algorithm uses Hough

408   transform and phase-coding (Yuen et al., 1990).

409   **RESULTS**

410   The results obtained in this study are divided into three main sections. The two first

411   sections correspond to porosity measurements made on the reference samples set only

412   (n= 30), either using the different helium gas porosimeters (i.e AccuPyc, AP-608 and

413   IHAP); or using the improved medical CT-scan methodology (Tab. 4). The third section

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414   presents the results obtained for the silurian core samples, using both IHAP and the

415   improved medical CT-scanning methodology (Tab. 5).

416   In addition to exact porosity values obtained from different instruments (AP-608,

417   AccuPyc, IHAP or CT-scan), several correlation ratios are then considered in these

418   results sections. Firstly, because the helium porosity measurements made using the IHAP

419   are considered as true values, the notion of absolute error is calculated with respect to the

420   helium porosity (Table 4-5). Second, the absolute error (AE) corresponding to the amount

421   of error in the porosity measurements is calculated, i.e. from the difference between the

422   porosity calculated using the improved CT-scan methodology and the porosity measured

423   using a conventional gas porosimeter. The R-squared (R^2), commonly used in classical

424   regression analysis (Rao et al., 1973), is calculated and represents a statistical measure of

425   how close the data are to the fitted regression line. Also known as the coefficient of

426   determination, R^2 ranges from 0 to 1. In the present work, R^2 is calculated to compare two

427   instruments or methodology evaluating the porosity. Finally, the root-mean-square error

428   (RMSE) is calculated. It corresponds to the standard deviation of the residuals (prediction

429   errors). Residuals are a measure of how far from the regression line data points are.

430   RMSE therefore represents how concentrated the data is around the line of best fit.

431   Applied to this paper, the line of best fit would correspond to a perfect match between

432   porosity measured with the CT-scan and that measured by AGAT laboratories using a

433   conventional helium gas porosimetry. RMSE was used to give an idea of how well the

434   CT-scan porosity matches the gas porosity obtained conventionally.

435   1) Helium porosity

436 The results allow for comparison of gas porosity obtained from three distinct instruments

437 and laboratories (Tab. 2; 4; Fig. 8A-C). All three methods are simple and rapid

438 techniques widely used to measure porosity on core samples. The porosity obtained from

439 Boyle's law gas porosimeters (Fig. 8A and B), IHAP, AP-608 and AccuPyc, shows linear

440 relationships with a R^2 coefficient ranging from 0.98 to 0.99 and a slope close to 1 (from

441 0.97 to 1.03). Accupyc and AP-608, with both measurement made at INRS, show the

442 smallest RMSE (0.43%) when compared with IHAP (0.8% for Accupyc and 0.94% for

443 AP-608). The larger differences between AP-608 and Accupyc (Fig. 8C) occur for

444 limestone samples (Indiana and Carthage) that are known to be genetically more complex

445 and spatially heterogeneous rocks (Galaup et al., 2012; Freire-Gormaly et al., 2015).

446 Therefore, differences in porosity evaluation were expected. The same trend is observed

447 when comparing IHAP with Accupyc and AP-608. This specific carbonate sample has a

448 highly irregular surface (Fig. 9), with visible vugs and dissolved bioclasts on the exterior

449 surface. Rock texture at surface can affect basic physical measurements such as length,

450 particularly those made with a caliper, and in turn, can induce uncertainty in porosity

451 estimation. Based on these comparison, results from sandstone samples appears to be

452 more consistent. Analysis obtained from AGAT Laboratories were chosen as the most

453 reliable since this private and independent laboratory runs routine petrophysical

454 properties analyses (including helium gas porosimetry) for the private oil and gas sector

455 as well as for academic and governmental research.

21

456   2) CT-scan porosity - reference samples

457   CT-scan porosity results are compared to IHAP only as the later values were validated

458   using the two different instruments and can therefore be considered as a reference (Fig.

459   10). The porosity obtained by the IHAP and CT-scan method (n=30) shows a linear

460   relationships with a R^2 coefficient of 0.99 and a slope close to 1 (0.91). When comparing

461   CT-scan-IHAP with AP-608-IHAP, RMSE is more than two times higher (Fig. 8A). This

462   difference is significant and tends to increase as porosity increases (Fig. 10). That can be

463   explained by the fact that more porosity means more water present inside the sample thus

464   a stronger beam hardening artefact variation. It leads to a larger underestimation of the

465   porosity as beam hardening creates a greater underestimation of the density in the

466   saturated state data.

467   A correction factor (1/0.91) was calculated using linear regression to minimize that

468   difference (Fig. 10). This correction brings the RMSE down to 0.54% (Fig. 11), which is

469   lower than AP-608 versus IHAP RMSE value (Fig. 8A), but slightly higher than AccuPyc

470   versus AP-608 RMSE (Fig. 8C). This correction factor was applied to all data points and

471   for all subsequent analyses. As already experienced by Ketcham and Iturrino (2005) ,

472   some voxels have estimated porosity values below 0 % (and above 100 %), due to image

473   noise and possible remaining misfit between data sets.

474   When considering each lithology type (Fig. 12), the CT-scan method seems to be less

475   robust with dolomite rock samples, where R^2 decreases down to 0.83 (Fig. 12C) and

476   RMSE is slightly higher. More data points would be needed to fully analyze these

477   relationships. The influence of porosity range has then been considered (Fig. 13) and

478   porosities values obtained using medical CT appears to be equally valid for the entire

22

479   range of porosities tested (from 1.5 to 34%), with R^2 around 0.98 (Fig. 13) and RMSE

480   close to 0.5. The possible influence of core diameter was also investigated.

481   When comparing porosity results of $1^{1/2}$" and $1^{7/8}$" core diameter (Fig. 14), R^2 are very

482   similar, with 0.99 (Fig. 14A) and 0.98 (Fig. 14B) respectively. However, RMSE increases

483   from 0.43 to 0.72 %, from small to large diameter which seems to indicate that, even after

484   linear correction, CT-scan method seems to produce more reliable porosity estimation for

485   smaller diameter samples.

486   Lastly, CT-scan porosity results using doping agent are also compared with IHAP (Fig.

487   15) and show a linear relationship with a R^2 coefficient of 0.99 and RMSE equals to

488   0.70%. These results are comparable to those obtained with water saturation and the

489   benefits of using NaI seems less important than initially expected.

490   *3)* CT-scan porosity – Silurian core interval

491   The selected HTD core interval (Fig. 4A) was subsampled and a 1.8 m continuous section

492   was scanned using the core-flooding setup. The interval actually corresponds to 18 core

493   subsamples (see Fig. 4 and Fig. 16). Within this 1.8 m interval, five isolated samples

494   were also sent to AGAT laboratories to validate locally the CT-scan porosity values (Fig.

495   4B; namely CSI-2, 3, 10, 12 and 16). Small gaps between core sub-samples were taken

496   into consideration and depths were corrected to account for these gaps. Different

497   statistical profiles describing porosity were generated (Fig. 16) with a spatial resolution of

498   0.6 mm. Mean porosity profile and heterogeneity profile indicate that porosity is lower in

499   the upper part of the depth interval but more heterogeneous, with a rather sharp transition

500   between subsamples CSI-7 and CSI-8 (Fig. 16). Another interesting observation is the

23

501   occurrence of increased porosity intervals (with values higher than 10%) that seems to be

502   limited to 10-15 cm thick interval (see for example within CSI-12 or 18). Such

503   information would be completely missed if considering discrete samples only for helium

504   gas porosity measurements. In addition to provide an average porosity value, CT-scan

505   porosity dataset also provides valuable spatial information about the porosity within

506   samples and allows porosity visualization in 3D (Fig. 17). Looking at diverse 3D views

507   from each sample individually, qualitative information is added, such as porosity

508   distribution vertically and horizontally, at the centimetric scale. For instance, within

509   sample CSI-2, largest macropores are homogeneously distributed within the sample and

510   correspond to isolated vugs (Fig. 17A), whereas in sample CSI-5, largest macropores are

511   limited to specific areas of the sample, associated to oblique fractures (Fig. 17B).

512   **DISCUSSION**

513   1) Porosity interpretation

514   Reference samples

515   Overall porosity correlation is a bit better for sandstone samples (Fig. 12). However,

516   RMSE for all lithologies remain quite close (and all <0.7%). RMSE increases slightly

517   from SST to LST and then Dol, a trend thatcoincide with porosity data points grouped

518   more tightly (around 10% porosity approximately). The number of samples changes from

519   N=14 to N=6, which can further affect the RMSE estimation. Even though our study use

520   a large number of samples (n=32) compared to the available literature, it is obvious that

521   the number of samples influences the quality of the results in a statistical point of view. In

522   future works, we plan to extend porosity range for limestone and dolomite to have truly

523   comparable dataset. However, the better porosity correlation for sandstone could very be

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524 statistically valid and genetic in nature, and this well-known in the literature (Lucia,

525 2007; Bust et al., 2011; Victor et al., 2017). Carbonates and dolomites, because of their

526 chemical reactivity have more complex diagenetic history and hence porosity

527 distribution. Because of their physical properties and generally lower chemical reactivity,

528 clastic sediments have more homogeneous porosity distribution to the contrary of

529 carbonates in which heterogeneous distribution of calcitic, aragonitic and dolomitic

530 components (particle, cement) will lead to irregular distribution of reactive particles to a

531 specific fluid, and hence variable, even erratic, porosity development at the very fine

532 scale.

533 Silurian core interval

534 The porosity evaluation of the Silurian core interval using CT-Scan (Fig. 16) gives

535 clearly heterogeneous values as expected given the nature of samples. The core

536 encompasses a mix of lithologies that do not appear affected by fracture-controlled

537 hydrothermal fluid circulation (Fig. 3A) and intervals with diverse degrees of

538 hydrothermal alteration (see Fig. 3B). The whole core set is a mix of preserved

539 depositional limestone facies (non-porous low energy depositional environment

540 represented by lime mudstone and wave reworked porous bioclastic limestone) and

541 hydrothermally altered diagenetic facies.

542 The improved methodology using medical CT-scan allows mapping out the porosity at a

543 centimetric scale, something impossible to achieve through conventional approaches. The

544 added value of the improved methodology is well illustrated when comparing dry state

545 CT-scan images of two dolomitic samples with similar porosity value (around 9.5 %; Fig.

546   18; Table 4-5). A reference sample (SI-K-15A) presents circular to ovoid mesopores

547   mainly concentrated on the lower half of the specimen (Fig. 18A). Macro/mesopores

548   appear disconnected from one another, at least at the medical-CT scale resolution. The

549   core sample coming from the silurian hydrothermally altered interval is completely

550   different (Fig. 18B), even though core-averaged porosity value are very close.

551   Macropores are much larger and their distribution is highly heterogeneous. Such

552   qualitative information cannot be deduced from conventional helium porosity. In

553   addition, when these two samples are analysed by our improved methology, core-

554   averaged porosity values are different. The reference samples, Si-K-15A, has a lower

555   porosity estimates (8.31%) whereas the silurian sample hydrothermaly altered has a

556   higher porosity estimates (11.3%). Looking at the images (Fig. 18), the CT-scan, core-

557   averaged, porosity values appears to be more realistic.

558   2) Advantages of this new CT scan methodology

559   The average CT porosity value for each reference sample has been used to validate the

560   methodology but the ultimate goal is to access the spatial distribution of porosity and acquire

561   further information on porosity at the macro scale. To achieve that, 2D and 3D visualisation

562   can be performed on the dataset to qualitatively describe the porosity distribution.

563   Quantitative statistics can also be derived to better describe not only porosity values but also

564   its spatial distribution: pore concentration at specific levels, heterogeneity variation with

565   function of depth, etc.

566   The experimental setup developed in the present study is cost-effective and easy to handle

567   (Fig. 5), especially when compare to previous core-flooding experiments found in the

568   literature (Hove et al., 1987; Vinegar and Wellington, 1987; Withjack, 1988), often using

569 costly pressure vessels made of aluminium chamber and Teflon casing (i.e. Hassler type core

570 holder; (Karacan et al., 2003). Different from Ketcham and Iturrino (2005) , our current setup

571 presents no risk of fluid loss while saturation and scanning phases because samples stay

572 within the core holder at all times. Numerical realignment of dry and wet states allows to

573 perform samples saturation phase (a minimum of 72 hours duration) outside of the CT

574 scanner requiring the use of the CT facility for a very limited amount of time. The low costs

575 of the core holder (PVC or Plexiglas) and custom, 3D printed internal fittings (PLA), makes

576 it easy to design a decimetric core-holder and analyse several samples at the same time. As a

577 matter of fact, the cost-effectiveness of both setup and protocol allowed to perform CT-scan

578 based porosities determination on 30 isolated samples and a 1.6 m thick core section. This is,

579 to the best of our knowledge, the first attempt ever made to run core-flooding experiments

580 under CT on such a large number samples (Tab. 1; Fig. 1). The scan time is quite reasonable

581 (2 minutes for 3 repeated scan on a 10cm sample), data processing time is fast (6 minutes)

582 and the method is scalable to process tens of meters of core samples. The saturation process

583 was performed for a week as the maximum water saturation was desired but this process

584 could be optimized by adjusting the following variables: (1) degassing time, (2) vacuum and

585 water circulation time and (3) water circulation duration without vacuum (Fig. 6). The second

586 step takes most of the process time and might be reduced considering the porosity level of the

587 sample. It might also be desirable to spend more time on the step 3 as remaining air bubbles

588 shrink in size and thus allow more water in. Likewise, it might be more effective to perform

589 alternative vacuum during step 2, as this could split entrapped air into smaller bubbles which

590 could then be extracted from the sample.

591 Different approaches used to derive quantitative information from CT data needs calibration.

592 Converting HU to density profiles requires subsampling, volume and weight measurements

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593   (Boespflug et al., 1994; Amos et al., 1996; ASTM-E1441-11, 2011). Mineralogy and porosity

594   variation obtained from "CT dual energy" acquisition are results derived from effective

595   atomic number and density profiles, which in turn needs calibration from materials standards

596   to convert X-ray absorption measurement (Van Geet and Swennen, 2001; Walls and

597   Armbruster, 2012; Lopez et al., 2016). Calibration is a non-trivial process and cumulates

598   errors from each additional required measurement. The proposed approach includes

599   calibration, as it requires air and fluid HU values, but is straightforward and taken in situ, for

600   every measurement made. Air HU value is measured while scanning samples at dry state and

601   fluid HU value during the saturated state scans.

602   No prior knowledge about mineralogy, density or porosity range is needed with the present

603   methodology to obtain a reliable porosity value (Figs. 10-16; Table 4-5). CT-scan porosities

604   obtained for dolomite samples are little less correlated with porosities values obtained by

605   conventional porosimetry, but overall lithology type still has a low influence on results (Fig.

606   12).

607   One of the main difference between porosities derived from CT-scan versus those given by

608   gas porosimeter is its independence from volume calculation. Gas porosimeters measure

609   grain volume. The porosity is then calculated using bulk volume of sample which is based on

610   linear measurements of samples with a caliper and the application of the appropriate

611   geometric formula. Therefore, this method is subject to human error and measurement error if

612   the sample is irregularly shaped (e.g. Fig. 9).

613   Lastly, working at medical CT scale represents an advantage when considering scanning time

614   and sample size. With our working parameters (Tab. 3), acquisition time is few seconds to

615   minutes, compare to micro or nano-CT where total data acquisition times span from hours to

616 ten of hours (Cnudde and Boone, 2013; Bultreys et al., 2016a) and where sample size is often

617 limited to only few millimeters (Pini and Madonna, 2016), which brings back the question of

618 the representativeness.

619   3) Limitations

620 One limitation of the proposed approach is the need to further correct beam hardening effects.

621 The original postulate was that artefacts such as beam hardening do not need to be corrected

622 because (1) the geometry is cylindrical (Pini and Madonna, 2016) and (2) remains constant

623 trough flooding experiment, i.e. beam hardening effect would be canceled out via the

624 subtraction of wet and dry datasets. However our results have shown that the difference

625 increases linearly as the porosity increases (Fig. 8). More porous samples (therefore

626 associated with the greatest difference between the two states), are associated with lower

627 correlations with gas-measured porosity values. Nonetheless, our current dataset suggests that

628 a simple linear correction seems to account well for the residual influence of beam hardening

629 (Fig. 10-11).

630 Cylindrical samples ease the selection of a versatile and low cost core holder material that

631 would then accommodate a large range of sample diameter. However the restrictive

632 geometry itself could be seen as a limiting factor. Without proper and full beam

633 hardening correction, the analysis of randomly shape rock fragments and other sample

634 geometry remains difficult under CT-scan. At best, qualitative information could be

635 obtained from non-cylindrical specimen.

636   4) Perspectives on future work (s)

637 Future research efforts should explore whether the experimental protocol, the acquisition

638 parameters or the data analysis itself can be optimized by lithology type or porosity

639   range. Maybe one or two repeated scans would be enough and therefore could lower the

640   cost to some instances.

641   Another key point for further works is to test upscaling possibilities. In particular, the

642   calibration of wireline logs profiles using the CT-scan porosity profiles, instead of

643   discrete porosity values from plugs, is promising. CT-scan images can be correlated

644   directly with density well log because they both measure the amount of Compton

645   scattering, proportional to bulk density (Wellington and Vinegar, 1987). With the high

646   levels of heterogeneity inherent in carbonate reservoirs, correlation between low

647   resolution e-logs and high resolution, discrete, poro-perm measurements has been

648   debated (Delhomme et al., 1996; Tilke et al., 2006). The medical CT and its range of

649   investigation could well bridges the gap.

650   As already stated, the production of 3D porosity matrix images (e.g. Fig. 17) opens the

651   opportunity to produce 3D models and run numerical flow simulations, at the centimetric

652   or tens of centimeter scale. This would be of interest for many research fields such as oil,

653   gas and geothermal reservoirs, hydrogeology or CO_2 sequestration.

654   CT-scan images were only made at the initial (samples filled with air) and final stages

655   (samples filled with distilled water), therefore no information regarding transitional

656   saturation conditions, wetting characteristics of rocks or permeability were gained.

657   Compared to similar studies in this field of research (Vinegar and Wellington, 1987;

658   Wellington and Vinegar, 1987; Withjack, 1988), there is no gas/fluid front to track.

659   Furthermore, the current setup rely only on capillary forces to saturate the rock samples.

660   Said differently, no fluid forcing is applied. One can argue that porosity data alone are

661 insufficient. Two samples with similar porosity values can have significantly different

662 permeability. However, the current setup not only allows the assessment of an average

663 porosity value per sample, but also provides a 3D porosity matrix (Fig. 17). This in turn,

664 can be transformed into 3D models and used to run numerical flow simulations using

665 commercial software such as COMSOL. Such approach has already been tested with

666 micro-CT and medical CT measurements (e.g. Zaretskiy et al., 2010; Bultreys et al.,

667 2015). To our knowledge, this has not commonly been achieved on heterogeneous

668 carbonate samples, for which pore structure measurements are mostly based on mercury

669 injection (Galaup et al., 2012), eventually combined with micro-CT for low porosity

670 carbonate (Fusi and Martinez-Martinez, 2013). A lot of work has been done in terms of

671 fluid flow modelling and simulation (see Review papers by Meakin and Tartakovsky

672 (2009) ; Blunt et al. (2013) and references therein) and might be adapted to our (medical)

673 CT-scan porosity dataset.

674 **CONCLUSION**

675 This work developed an effective and practical method using medical-CT to reliably

676 estimate reservoir porosity for spatially heterogeneous material such as fractured or

677 dolomitized carbonates, incorporating recent advances in data correction.

678 (1) The in-house core-flooding setup is low cost, simple, and easy to operate. Several

679 individual core samples can be scanned simultaneously (dry and saturated), as well as

680 continuous core sections up to 1.5 m long. Scanning a sample in a dry state and saturated

681 state, performing a three-dimensional alignment and subtracting the two data sets allow

682 the construction of 3D porosity matrices.

31

683   (2) Based on a set of reference core material, this study illustrates the relationship

684   between porosity assessed by CT-scan against the ones obtained by conventional gas

685   porosimetry techniques. A strong correlation is observed between both techniques so that

686   the current CT-scan methodology appears to be a reliable and acurate way to estimate

687   fine-scale variations of porosity for the main types of sedimentary rocks, in a wide range

688   of porosity value.

689   (3) This consistency opens up the possibility to extend porosity assessment beyond gas

690   porosimetry, particularly for heterogeneous carbonate samples. The added value of the

691   porosity measurement by CT-scan is the generation of 3D images of pore network,

692   allowing to assess spatial attributes of macropores, their distribution and connectivity.

693   (4) Last, but not least, the CT-scan method allows the construction of continuous porosity

694   profiles that are well correlated with discrete helium gas porosity values.

695   Millimetric/centimetric scale data are rarely available in subsurface datasets and reliable

696   continuous porosity measurement at this scale is a step forward in the understanding of

697   reservoir properties.

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1105 **FIGURES CAPTIONS**

1106 **TAB. 1** List of references samples used for the present work. In the sample name, the

1107 petroleum core sample provider is indicated, with K standing for Kocurek Industries Inc

1108 and C standing for Cleveland Quarries. All basic samples measurements were performed

1109 at the LOG, using a digital caliper and a precision scale. BE stands for Berea sandstone;

1110 SC stands for Scioto sandstone; BO stands for Boise sandstone, NU stands for Nugget

1111 sandstone; IN stands for Indiana sandstone; CA stands for Carthage Marble (= Burlington

1112 Limestone); GE stands for Guelph dolomite; SI stands for Silurian dolomite.

1113 **TAB. 2** Comparison of methods used in the present work to estimate porosity.

1114 **TAB. 3** Summary of CT-scanner parameters values for both acquisition and reconstitution

1115 stages. kV stands for kilovoltage, mAs for milliampere-second, F.O.V. for field of view,

1116 and HU for Hounsfield Unit.

1117 **TAB. 4** Summary of results for reference core samples. The absolute error (AE)

1118 corresponds to the difference between the porosity calculated using the improved CT-

1119 scan methodology and the porosity measured using the conventional gas porosimeter

1120 IHAP.

1121 **TAB. 5** Summary of results for silurian core samples.

1122 **FIG. 1** Range of porosity tested for this work. Reference core samples correspond to eight

1123 different lithologies commonly used as test material in the petroleum industry, and cover

1124 a large range of porosity (2-5 to 28 %), namely Berea, Scioto, Nugget and Boise

1125 sandstones (SST), Indiana and Burlington limestones (LST), and Silurian and Guelph

1126 dolomites. For each lithology type, an expected porosity or porosity range was given by

1127 the vendor Kocureck Industries and these values are reported here.

1128 **FIG. 2** Petrographic attributes of reference core material. (A) Microphotograph of

1129 medium-grained Nugget sandstone sample, with moderate sorting and well-rounded

1130 grains. (B) Microphotograph of medium to coarse-grained Boise sandstone sample, with

1131 poor sorting. Quartz grains are angular (C-D) Microphotographs of fine-grained, well

1132 sorted Scioto sandstone. Quartz grains are subangular. (E) Microphotograph of Carthage

1133 marble limestone. This limestone is a well cemented, fossiliferous limestone with

1134 moderate to poor sorting. (F) Microphotograph of homogeneous fine grained Silurian

1135 dolostone within the Sayabec Formation.

1136 **FIG. 3**. Simplified geological map of the Témiscouata area in eastern Quebec (Canada)

1137 with the location of the Massé No 1 well (black star). The yellow star locates the position

1138 of an exhumed hydrocarbon field hosted in hydrothermal dolomite (HTD). The

1139 stratigraphic column to the left locates the Sayabec - Val Brillant interval deposited at the

1140 end of the first shallowing event (S1) and onset of the first deepening event (D1) in the

1141 Gaspé Belt. Stratigraphic details and basin evolution are found in Bourque et al. (1995).

1142

1143 **FIG. 4**. (A) Macrophotograph of silurian core interval used for this study. These

1144 continuous 4.5 meter long core section belong to the lower Silurian Sayabec Formation in

1145 Massé No. 1 well drilled within the Massé structure (Lower St-Lawrence river area,

1146 Québec). Core samples are 4.5 cm in diameter and their length ranges from 5 to 10 cm

1147 (approximately). (B) Macrophotograph of five Silurian core samples, chosen to illustrate

1148 the natural heterogeneity of this interval along depth. Each sample is briefly described

1149 and porosity is assessed from macroscopic observations on the surface.

46

1150   **FIG. 5** Schematic diagram of the core flooding experimental setup for porosity

1151   measurement. The water tank is a closed reservoir with a 4 L total volume. To

1152   accommodate meter long core section, four chamber are set in parallel.

1153   **FIG. 6** Workflow chart illustrating the successive steps involved in the present work and

1154   separated in three groups: experiment, CT-scanning acquisition and processing.

1155   **FIG. 7** (A) Impact of noise level on porosity calculation and its uncertainty level (adopted

1156   from Pini and Madonna (2016)). (B) Axial CT-scans with decreasing resolution. This

1157   illustrates how fine structures (such as fractures) could remain undetected if the

1158   resolution is too low. The spatial resolution was then set to 0.1 x 0.1 x 0.6 mm.

1159   **FIG. 8** Statistical comparison of three gas porosity measurement techniques. (A) IHAP

1160   versus AP-608 with n = 20, (B) IHAP versus AccuPyc with n = 10, and (C) AP-608

1161   versus AccuPyc, with n = 10.

1162   **FIG. 9** Indiana limestone sample (IN-C-178B) with its highly irregular surface.

1163   Macropores and core damages are abundant on the external surface which produce an

1164   imprecise total volume calculation using caliper and could induce the outlier data point

1165   (see Fig. 6; Fig. 9A).

1166   **FIG. 10** Statistical comparison of CT-scan porosity measurement technique against

1167   conventional gas porosity technique (IHAP). The outlier results (white star) corresponds

1168   to carbonate sample IN-C-178B, and was not considered for regression. For further

1169   analyses and subsequent figures, the slope of the regression line is used as a correction

1170   factor.

1171   **FIG. 11** Statistical comparison of CT-scan porosities against IHAP after correction. The

1172   correction factor used (1/0.91) intends to correct beam hardening effect. The outlier

1173   carbonate sample (IN-C-178B) and was not considered for regression.

1174   **FIG. 12** Lithology influence on the correlation between CT-scan porosity method and

1175   conventional IHAP. Note that all data point used are corrected values. (A) Data points for

1176   sandstones (n=14); (B) data points for limestones (n=10) and (C) data points for

1177   dolomites (n=6). Indiana limestone outlier (IN-C-178B) was not considered for

1178   regression.

1179   **FIG. 13** Porosity range influence on the correlation between CT-scan porosities and

1180   conventional IHAP. Note that all data point used are corrected values. Low porosity

1181   range is defined as porosity values lower than 15 % (n=10); and high porosity range is

1182   defined as porosity values higher than 15% (n=19). Indiana outlier (IN-C-178B) was not

1183   taken in consideration for regression. R^2 for both ranges of porosity values reaches 0.98,

1184   and RMSE are rather low, close to 0.5.

1185   **FIG. 14** Core diameter influence on the correlation between CT-scan porosities and

1186   conventional IHAP, with (A) 1½" diameter core samples and (B) $1^{7/8}$"diameter core

1187   samples.

1188   **FIG. 15** Statistical comparison of CT-scan porosities obtained with doping agent (NaI)

1189   against IHAP porosities. Note that all data point used are corrected values. Two outlier

1190   samples (not shown) were not considered for regression. Both outlier correspond to

1191   Indiana limestone samples, and one of them is IN-C-178B (Fig. 8).

1192   **FIG. 16** Continuous porosity profiles obtained for a 1.8 meters thick section of the lower

1193   Silurian hydrothermal dolomites. The average porosity for this entire section is 3.38%.

1194 The continuous porosity values are compared to discrete helium gas porosity values

1195 obtained at the AGAT laboratories, and CT-scan porosity value average per subsamples

1196 (CSI-3, CSI-6, CSI-10, CSI-12 and CSI-16). The different profiles were plotted using IP

1197 software. A bell (Gaussian) filter was applied to the mean CT-scan porosity values. HI

1198 (heterogeneity index) and CV (coefficient of variation) parameters are adopted from

1199 Caliskan and Shebatalhamd (2017)

1200 **FIG. 17** Examples of 2D views illustrating connected porosity matrices obtained for two

1201 specific subsamples within the HTD interval. Greyscale indicates porosity, from 0 %

1202 (black) to 100% (white). (A-B) correspond to coronal and sagittal mean intensity

1203 projections views (MIP) respectively of the sample CSI-2 Sample CSI-5 MIP views are

1204 shown in the same manner in (C-D). See Fig. 15 for samples location. Provided as

1205 supplementary material, 360° rotation movies of these two samples were made using

1206 Dragonfly software.

1207 **FIG. 18** Examples of coronal CT images from (A) a reference core sample, namely SI-K-

1208 15A and from (B) one sample from the HTD interval, namely CSI-12 (see Fig. 16 for its

1209 location along the porosity profile). Both dolomite samples have similar porosity values

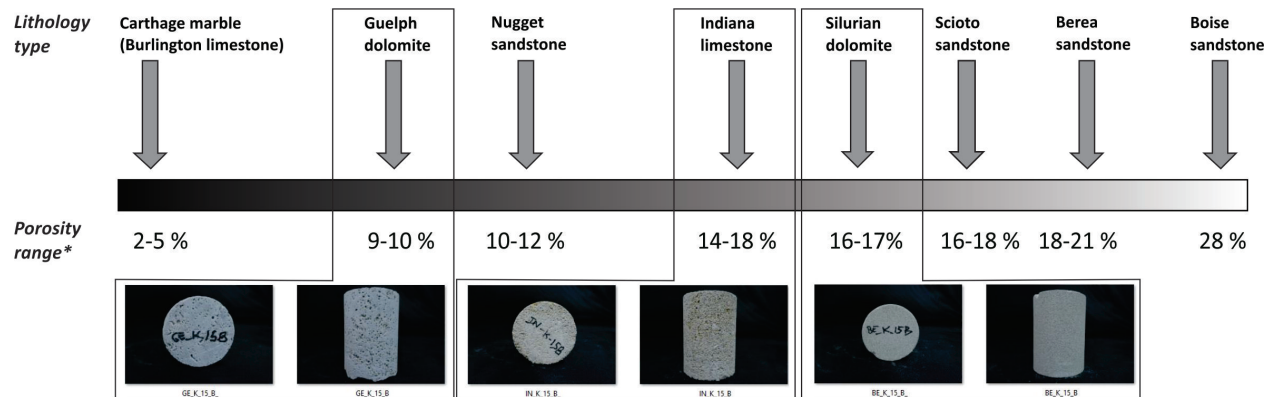1210 obtained by IHAP, i.e. 9.5% and 9.32 % respectively (see Table 4 and 5).

Location	Number of specimen	Lithology type	Average porosity (from litterature)	References
Ohio (USA)	8	Berea SST	20%	Winkler, 1983; Churcher et al., 1991; Hart and Wang, 1995; Boon et al., 2017
Utah and Wyoming (USA)	2	Nugget SST	up to 25%	Lindquist, 1988
Idaho (USA)	2	Boise SST	28-30%	Winkler, 1983
Ohio (USA)	2	Scioto SST	12%	Holder et al., 2001; Bose et al., 2014
Indiana (USA)	8	Indiana LST	13%	Musselman, 1967; Schmidt and Huddle, 1977; Churcher et al., 1991; Hart and Wang, 1995; Boon et al., 2017
Ohio (USA)	2	Carthage LST	1.5%	Musselman, 1967; Martin, 1968
Ohio (USA)	2	Silurian dolomite	14-20%	Islam et al., 2018
Ohio (USA) and Ontario (Canada)	4	Guelph dolomite	7-24%	Churcher et al., 1991

	AccuPyc	AP-608 porosimeter	AGAT Helium porosimeter	CT-scan
Sample length (cm)	2.54	2.54 to 10.16	2.54 to 7.62	up to 250
Sample diameter (cm)	2.54	2.54	2.54 or 3.81 or 5.08	up to 50
Injection pressure (psi)	20	200	100	*not applicable*

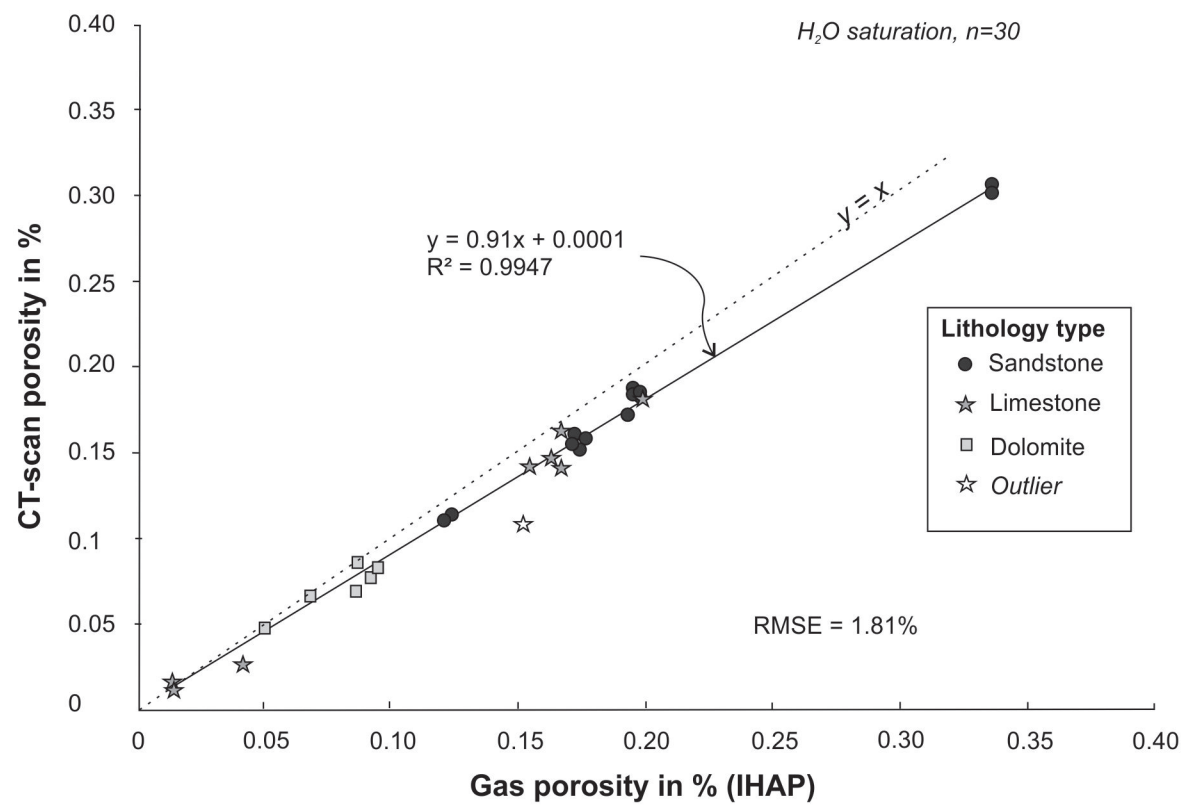|	AccuPyc	AP-608 porosimeter	AGAT Helium porosimeter	CT-scan
Sample length (cm)	2.54	2.54 to 10.16	2.54 to 7.62	up to 250
Sample diameter (cm)	2.54	2.54	2.54 or 3.81 or 5.08	up to 50
Injection pressure (psi)	20	200	100	*not applicable*

Acquisition parameters

	Reference samples	Natural core samples
kVp	140	140
mAs	700	350
Pitch	0.55	0.55
Collimation	20 x 0,6 mm	20 x 0,6 mm

Reconstruction parameters

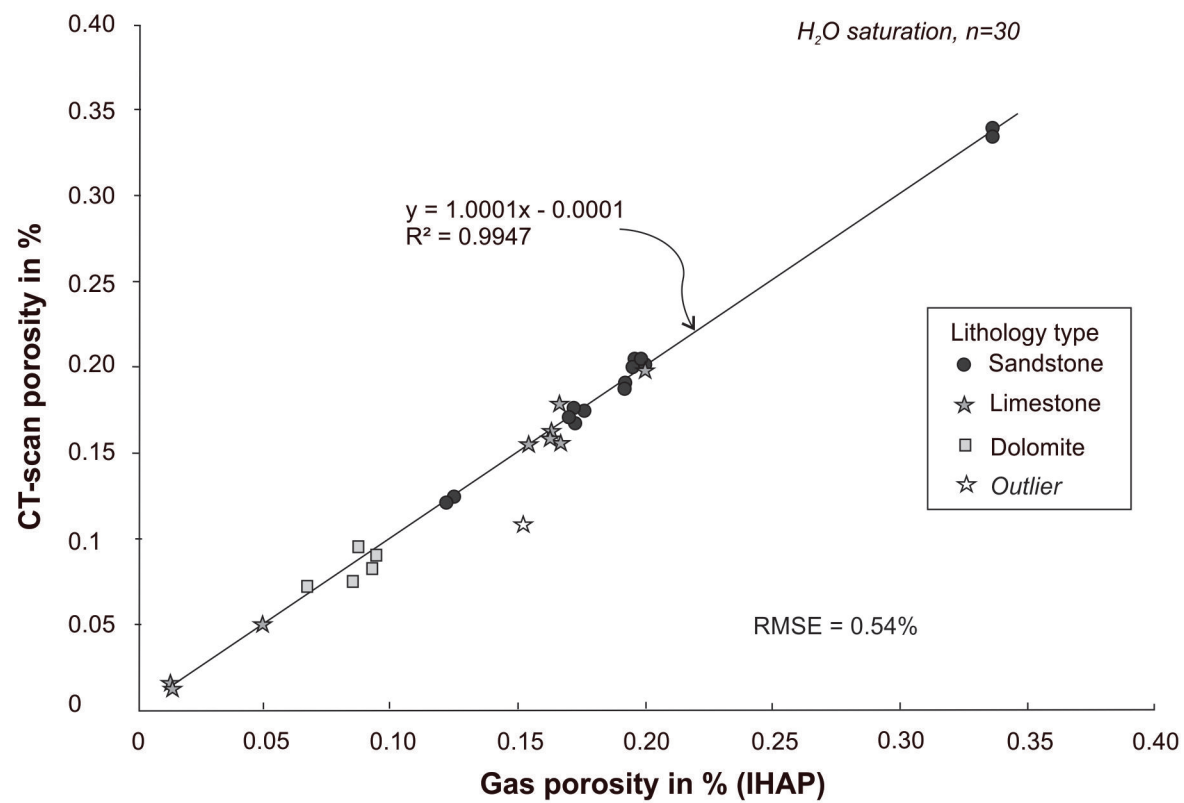|	Reference samples	Natural core samples
Filter	H70h	H70h
F.O.V	60 mm	55 mm
Pixels spacing	0.1172 x 0.1172	0.1074 x 0.1074
Slice thickness	0.6 mm	0.6 mm
HU scale	normal	normal
Focal spot	1.2 mm	1.2 mm

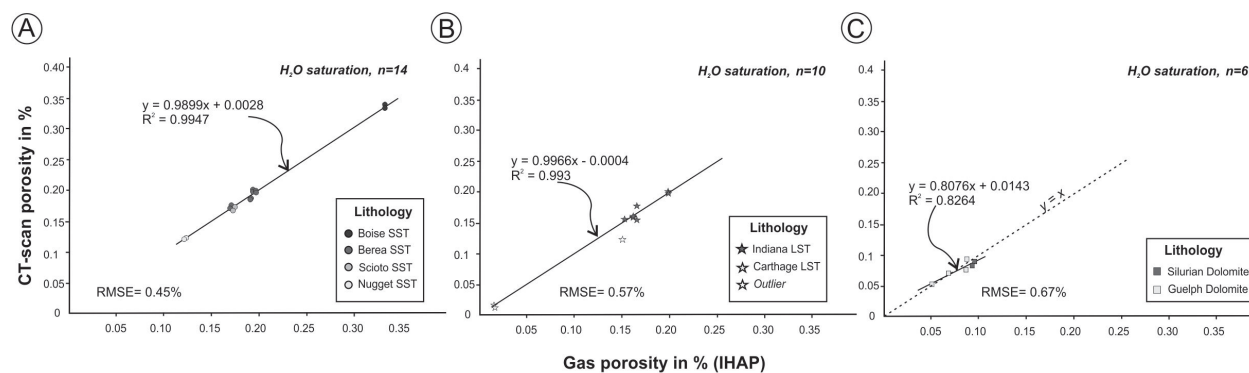| Sample attributes					Measured porosity (this work)						Reference porosity	
Sample name	Length (mm)	Diameter (mm)	Weight (g)	Lithology	AccuPyc	AP-608	IHAP	CT-scan (H2O)	CT-scan (NaI)	Absolute error	Littérature	Core vendor
BE_C_15_A	9.9	3.8	236.00	Berea SST	n/a	20.30%	19.80%	20.02%	n/a	0.22%		
BE_C_15_B	5.2	3.8	121.70	Berea SST	20.16%	20.31%	19.80%	20.35%	17.21%	0.55%		
BE_C_178_A	74.9	4.5	287.87	Berea SST	n/a	n/a	17.10%	17.12%	n/a	0.02%		
BE_C_178_B	75.2	4.5	290.12	Berea SST	n/a	n/a	17.20%	17.71%	12.75%	0.51%	20%	18-21%
BE_K_15_A	9.9	3.8	238.30	Berea SST	n/a	19.74%	19.30%	18.95%	n/a	0.35%		
BE_K_15_B	5.1	3.8	124.00	Berea SST	19.38%	20.25%	19.30%	18.86%	16.52%	0.44%		
BE_K_178_A	74.6	4.5	311.50	Berea SST	n/a	n/a	19.50%	20.38%	n/a	0.88%		
BE_K_178_B	75.5	4.5	315.28	Berea SST	n/a	n/a	19.50%	20.20%	16.14%	0.70%		
NU-K-15A	8.21	3.83	217.00	Nugget SST	n/a	13.27%	12.10%	12.20%	n/a	0.10%	up to 25%	10-12%
NU-K-15B	4.97	3.84	130.80	Nugget SST	13.63%	14.23%	12.30%	12.34%	10.57%	0.04%		
BO-K-15A	8.13	3.79	156.37	Boise SST	n/a	34.99%	33.60%	33.68%	n/a	0.08%	28-30%	128%
BO-K-15B	4.91	3.79	94.51	Boise SST	34.60%	34.36%	33.60%	33.19%	28.45%	0.41%		
SC-K-15A	8.13	3.82	199.80	Scioto SST	n/a	19.45%	17.40%	16.75%	n/a	0.65%	12%	16-18%
SC-K-15B	4.93	3.81	121.20	Scioto SST	18.90%	18.89%	17.60%	17.45%	14.82%	0.15%		
IN_C_15_A	9.90	3.77	248.50	Indiana LST	n/a	17.18%	16.30%	16.15%	n/a	0.15%		
IN_C_15_B	5.10	3.77	127.70	Indiana LST	16.61%	17.59%	16.30%	16.04%	12.77%	0.26%		
IN_C_178_A	74.36	4.50	294.95	Indiana LST	n/a	n/a	15.40%	15.58%	n/a	0.18%		
IN_C_178_B	76.31	4.50	304.64	Indiana LST	n/a	n/a	15.20%	11.46%	10.60%	3.74%	13%	14-18%
IN_K_15_A	9.80	3.76	233.30	Indiana LST	n/a	20.45%	19.90%	19.88%	n/a	0.02%		
IN_K_15_B	5.20	3.76	123.20	Indiana LST	18.85%	21.01%	19.90%	19.97%	15.98%	0.07%		
IN_K_178_A	74.38	4.50	325.99	Indiana LST	n/a	n/a	16.70%	17.88%	n/a	1.18%		
IN_K_178_B	75.50	4.50	331.30	Indiana LST	n/a	n/a	16.70%	15.53%	7.75%	1.17%		
CA-K-15A	8.07	3.81	242.70	Carthage LST	n/a	2.76%	1.40%	1.37%	n/a	0.03%	1.50%	2-5%
CA-K-15B	5.01	3.818	150.4	Carthage LST	2.82%	3.34%	1.40%	1.51%	0.57%	0.11%		
SI-K-15A	8.05	3.83	236.10	Silurian Dol	n/a	9.69%	9.50%	9.11%	n/a	0.39%	14-20%	16-18%
SI-K-15B	4.99	3.84	146.20	Silurian Dol	10.14%	10.09%	9.20%	8.38%	6.47%	0.82%		
GE_K_15_A	8.95	3.78	260.00	Guelph Dol	n/a	8.96%	8.60%	7.65%	n/a	0.95%		
GE_K_15_B	5.14	3.78	147.70	Guelph Dol	8.94%	10.63%	8.70%	9.49%	7.03%	0.79%	7-24%	9-10%
GE_K_178_A	66.91	4.50	355.17	Guelph Dol	n/a	n/a	5.00%	5.22%	n/a	0.22%		
GE_K_178_B	76.13	4.50	397.71	Guelph Dol	n/a	n/a	6.80%	7.32%	5.24%	0.52%		

	Sample attributes		Measured porosity (this work)	
Sample name	Length (cm)	Diameter (mm)	CT-scan (H2O) *corrected*	IHAP
CSI-1	13.0	4.5	7.76%	n/a
CSI-2	9.0	4.5	7.03%	n/a
CSI-3	5.5	4.5	**2.51%**	**2.10%**
CSI-4	12.5	4.5	1.93%	n/a
CSI-5	11.0	4.5	2.76%	n/a
CSI-6	6.0	4.5	**2.42%**	**1.90%**
CSI-7	5.0	4.5	1.43%	n/a
CSI-8	18.0	4.5	6.82%	n/a
CSI-9	6.50	4.5	5.73%	n/a
CSI-10	7.00	4.5	**4.54%**	**4.20%**
CSI-11	17.00	4.5	3.99%	n/a
CSI-12	11.00	4.5	**9.36%**	**11.30%**
CSI-13	5.00	4.5	4.35%	n/a
CSI-14	11.00	4.5	8.89%	n/a
CSI-15	4.00	4.5	7.43%	n/a
CSI-16	6.00	4.5	**2.53%**	**3.00%**
CSI-17	4.00	4.5	3.60%	n/a
CSI-18	9.50	4.5	2.92%	n/a

Lithology type

Carthage marble (Burlington limestone) | Guelph dolomite | Nugget sandstone | Indiana limestone | Silurian dolomite | Scioto sandstone | Berea sandstone | Boise sandstone

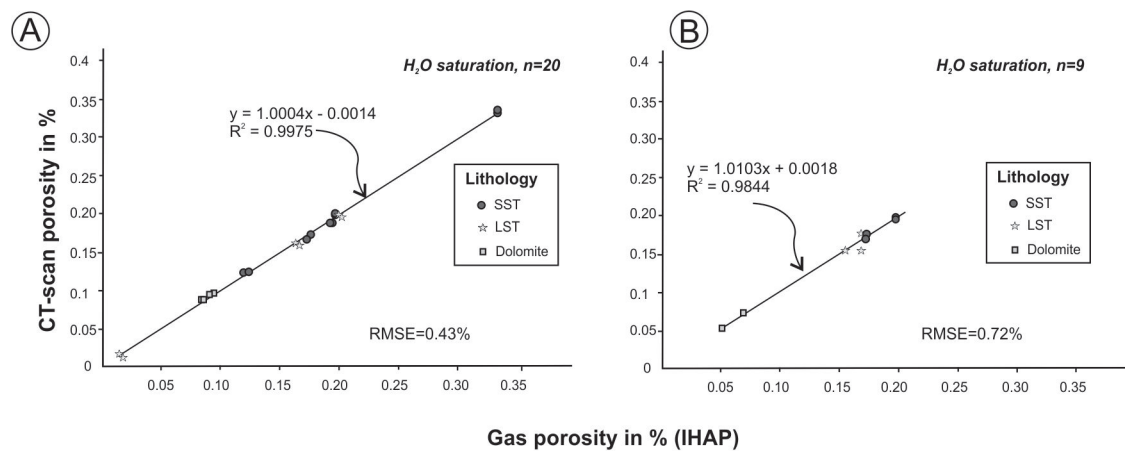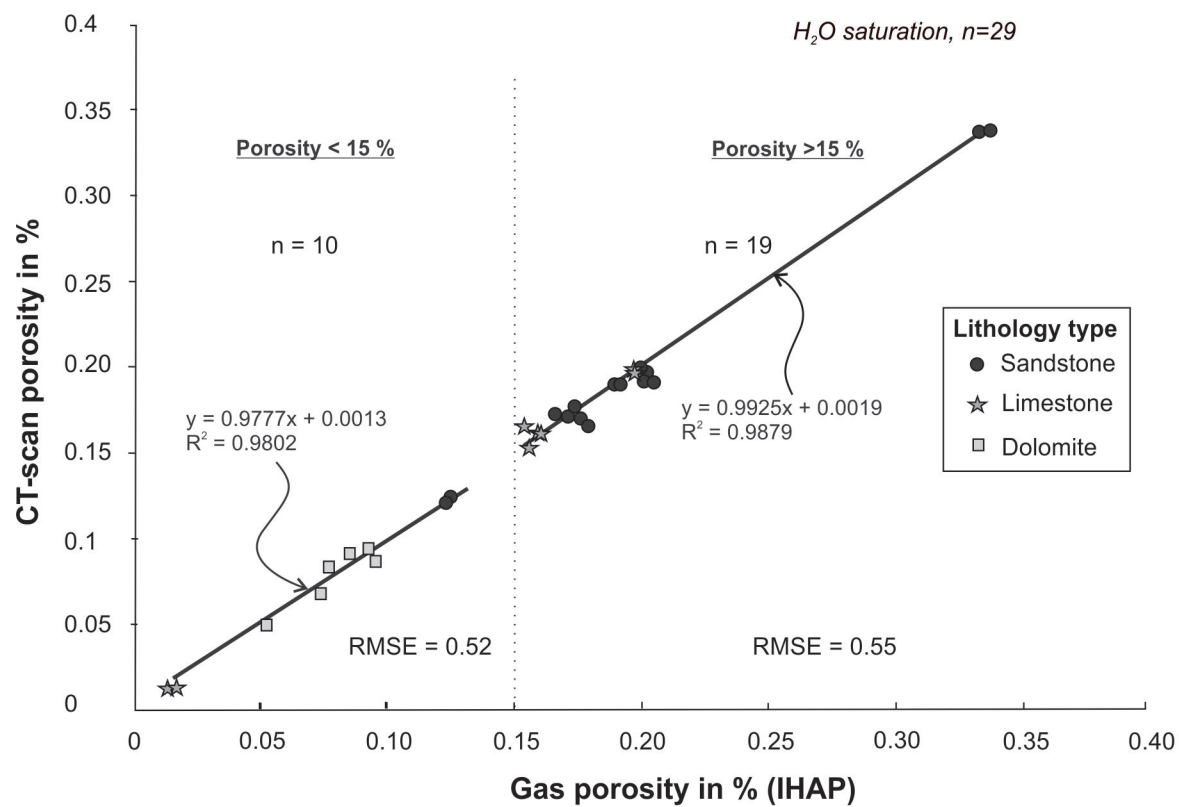**Porosity range***

2-5 % | 9-10 % | 10-12 % | 14-18 % | 16-17% | 16-18 % | 18-21 % | 28 %

source Kocurek Industries Hard Rock Division (https://kocurekindustries.com)

H_2O saturation, $n=30$

$y = 1.0001x - 0.0001$
$R^2 = 0.9947$

RMSE = 0.54%

Lithology type
● Sandstone
☆ Limestone
▢ Dolomite
☆ *Outlier*

CT-scan porosity in %

Gas porosity in % (IHAP)

A   *H₂O saturation, n=14*

CT-scan porosity in %

y = 0.9899x + 0.0028
R² = 0.9947

RMSE= 0.45%

Lithology
- Boise SST
- Berea SST
- Scioto SST
- Nugget SST

B   *H₂O saturation, n=10*

y = 0.9966x - 0.0004
R² = 0.993

RMSE= 0.57%

Lithology
- Indiana LST
- Carthage LST
- *Outlier*

C   *H₂O saturation, n=6*

y = 0.8076x + 0.0143
R² = 0.8264

y = x

RMSE= 0.67%

Lithology
- Silurian Dolomite
- Guelph Dolomite

Gas porosity in % (IHAP)

A

H_2O saturation, n=20

CT-scan porosity in %

$y = 1.0004x - 0.0014$
$R^2 = 0.9975$

Lithology
● SST
☆ LST
□ Dolomite

RMSE=0.43%

B

H_2O saturation, n=9

$y = 1.0103x + 0.0018$
$R^2 = 0.9844$

Lithology
● SST
☆ LST
□ Dolomite

RMSE=0.72%

Gas porosity in % (IHAP)

H₂O saturation, n=29

Depth	Subsample	Porosity profiles	Discrete porosity measurements	Statistical parameter profiles	Macrophotograph

Porosity profiles legend:
- Discrete IHAP_porosity (0 – 25)
- Mean CT_Porosity (0 – 25)
- Filtered_Mean_CT_Porosity (0 – 25)

Discrete porosity measurements:
CT-scan porosity *   Gas porosity (IHAP)
* average value per subsample

Statistical parameter profiles:
- HI (0 – 500)
- CV (0 – 30)

Depth	Subsample	CT-scan porosity	Gas porosity (IHAP)
	CSI-1		
	CSI-2		
1558.5	CSI-3	2.26%	2.1%
	CSI-4		
	CSI-5		
	CSI-6	1.84%	1.9%
	CSI-7		
1559			
	CSI-8		
	CSI-9		
	CSI-10	2.76%	4.2%
1559.5	CSI-11		
	CSI-12	9.54%	11.3%
	CSI-13		
	CSI-14		
1560	CSI-15		
	CSI-16	2.21%	3.0%
	CSI-17		
	CSI-18		

Vacuum
pump

Diaphragm
pump

Parallel PVC chambers

Core samples

Core samples

Core samples

Core samples

distributor

collector

Reservoir

Legend

Main valve

Pump

Quick connector

Core holder internal design

core samples

Sample(s) in oven for 48h at 108°C

Sample(s) in chamber(s)

3 scans in 'dry' state

'Dry' state scans averaged

Connexion to both pumps

+

(1) Water tank and samples under vacuum (70 cm/Hg) for 24h

(2) Core-flooding with constant fluid circulation for a minimum of 48h and under vaccuum

(3) Fluid circulation at ambiant pressure for 24h (no vacuum)

3 scans in 'saturated' state

'Saturated' state scans averaged

Legend

Experiment

CT-scan Acquisition

Processing

Saturated and dry 3D datasets are registered

'Substraction of 'dry' and 'saturated' density matrices

Porosity and 3D pore network

Statistical parameters associated with porosity distribution

Continuous porosity profile

3D connected porosity matrices

Average porosity for discrete samples

A

$y = 1.0114x - 0.0127$
$R^2 = 0.9955$
RMSE = 0.80%

y = x

Lithology type
● Sandstone
★ Limestone
□ Dolomite

n = 20

Gas porosity in % (IHAP)

Gas porosity in % (AP-608)

B

$y = 1.0139x - 0.0081$
$R^2 = 0.9924$
RMSE = 0.94%

y = x

Lithologiy type
● Sandstone
★ Limestone
□ Dolomite

n = 10

Gas porosity in % (IHAP)

Gas porosity in % (AccuPyc)

C

$y = 0.9726x + 0.0113$
$R^2 = 0.9917$
RMSE = 0.43%

y = x

Lithology type
● Sandstone
★ Limestone
□ Dolomite

n = 10

Gas porosity in % (AP-608)

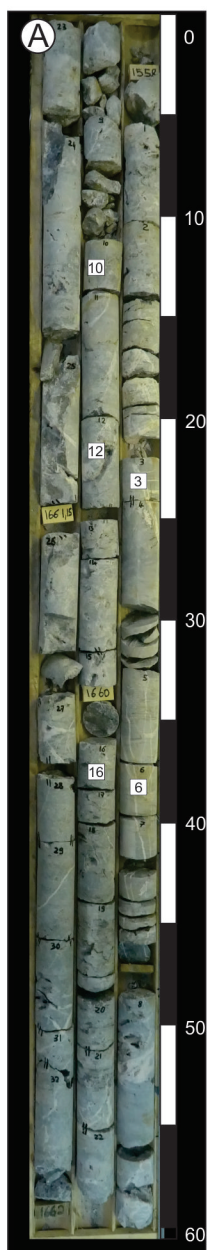Gas porosity in % (AccuPyc)

Silurian sample #3
Description
Fine-grained limestone with horizontal and obliques fractures and veins.

Porosity
Porosity wasexpected to be relatively low, and associated with unsealed fractures.

Silurian sample #6
Description
Fine-grained, laminated limestone with vertical and obliques fractures and veins.

Porosity
Open porosity is visible on sample surfacem associated with oblique fractures. However, porosity is expected to be low.

Silurian sample #10
Description
Massive dolomitized limestone with original depositional texture not visible at the macroscale.

Porosity
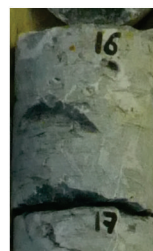Thin cracks associated with visible porosity (intergranular porosity?).

Silurian sample #12
Description
Massive dolomitized limestone largely affected by centimetric fractures and dissolution. Original depositional texture is not preserved.

Porosity
Open porosity is visible on surface and associated with large, centimetric fractures.

Silurian sample #16
Description
Bioclastic limestone with millimetric to plurimillimetric open vugs, often associated with moldic porosity.

Porosity
Open porosity is visible on surface sample as dissolved molds.

Highlights

(1) Combined core-flooding setup and medical-CT give porosity for heterogeneous material.

(2) Reference core material were tested and included 7 different lithologies

(3) The new CT methodology developed strongly correlates with conventional gas porosimetry.

(4) 3D porosity matrices and continuous porosity profiles at submillimetric scale are produced.